

**Use of IR Spectroscopy, X-Ray Diffraction and Pectrografic Analysis to evaluate  
Structural changes in Alumophosphate Compositions at Creeping<sup>1</sup>**

U. Shayachmetov<sup>2,4</sup>, R. Shayachmetov<sup>2</sup> and Ion Dranca<sup>3</sup>

---

<sup>1</sup> Paper presented at the Fourteenth Symposium on Thermophysical  
Properties, June 25-30, 2000, Boulder, Colorado, U.S.A.

<sup>2</sup> State Enterprise «Bashkir Scientific Research and Design Institute for  
Industry of Building Materials»

<sup>3</sup> Guest scientist from the Institute of Chemistry, Chisinau, MD-2028  
Moldova

<sup>4</sup> To whom correspondence should be addressed

ABSTRACT

The present study is an attempt to evaluate the structural changes observed in ceramic compositions on the basis of aluminum oxide and phosphate binders ( $H_3PO_4$  or ACPB) at creeping with the use of IR spectroscopy, X-ray Diffraction and Pectrographic Analysis, where ACPB -aluminumchromphosphate binder. The study reports the dependence of structural changes on the composition of the compounds, the type of their phosphate binder, the temperature of processing and the test conditions at creeping.

On the basis of the test results and the data obtained elsewhere [1-7] it was surmised that deformation process of phosphate compositions at high temperatures and loads is connected with a change of physicochemical characteristics of the material, and as its consequence with the change of its structure. It was revealed that the mechanism of deformation of alumophosphate material at creeping depends primarily on the change in the matrix structure that cements the grains of electrocorundum in the material.

**KEY WORDS:** Aluminum Phoshate Composition, Creeping, IR-spectroscopy, Pectrographic Analysis, X-ray Diffraction

## 1. INTRODUCTION

The chemical technology of phosphate materials is one of the new trends in the field of material study, the development of which allowed to produce a wide range of effective materials on the basis of the system-oxide metal phosphate binder. Such systems are rewarding objects for research and development of the fire proof materials and objects on their basis. The use of the latter has considerably prolonged the service term of lining in heating installations [8].

Phosphate materials as well as many other ceramic materials are brittle, and yet at high temperatures plastic deformation and sometimes even rapid destruction is observed. The peculiarities of such processes and high temperature creeping of ceramic materials are studied in detail elsewhere [1-7].

Alongside with study of dependence of creeping on temperature, load, reaction time, deformation processes of ceramic and fire proof materials have been studied. The dependence of deformation creeping processes on the types of chemical binding and the structure of elementary cell has been shown [5].

There is enough data in literature on the processes of hardening and structure formation of alumophosphate materials with the use of IR spectroscopy [9-19; 21-24]. As far as the interaction of aluminum oxide with orthophosphoric acid is concerned the data is numerous but contradictory [20, 22]. There is certain information on deformation at creeping of phosphate materials [21], but there is no data in literature on the study of this process with the use of physical chemistry.

The objective of the work reported herein is to study the physicochemical processes of hardening and structure forming that occur in binding systems and compositions on the basis of aluminum oxide and phosphate binder depending on the initial components in the compositions, the type and the temperature of treating the binder, as well as the study of structural changes after their high temperature deformation at creeping by IR-spectroscopy, X-ray Diffraction and Pectographic analysis.

## 2. MEASUREMENTS

## 2.1. Specimens

To prepare samples of binders and compositions of different structures (according to the table) there were used -  $\text{Al}_2\text{O}_3$ , electrocorundum N $\text{\textcircled{5}}$ 0 and N $\text{\textcircled{6}}$ , fine ground unhydrated alumina as initial powder components (FGAN). As hardening liquid 85%  $\text{H}_3\text{PO}_4$  or alumo-chromo-phosphate binder, which is a solution of different aluminum and chrome phosphates with the common formula  $\text{Al}_2\text{O}_3 \cdot x\text{Cr}_2\text{O}_3 \cdot y\text{P}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ , was used. The samples were prepared in the following way: dry components were mixed with the phosphate binder; the samples were formed from the prepared mass and thermally treated at 300, 900 and 1500 $^{\circ}\text{C}$ . Alumo-phosphate samples which were preliminary treated at 300 $^{\circ}\text{C}$  were additionally tested for creeping on a unit designed in GUP «BashNIPiIstrom» [21].

## 2.2. Procedure

Infrared (IR) Absorption Spectra were obtained using M-80 Spectrophotometer (Germany). Their infrared binder and composition spectra were read on a spectrum meter M-80 (Germany). Grinding in an agate mortar and mixing with vaseline oil prepared the samples. The crystal solidification product content was defined on a diffractometer DRON-3 with  $\text{Co-K}_{\alpha}$  radiation (iron filter). Petrographic analysis was conducted using of MIN-8 microscope.

## 3. RESULTS

To determine the character of structural changes in compositions after their deformation at creeping there were primarily analyzed the processes of structure forming in viscous systems and compositions based on aluminum oxide and phosphate binder at heating the samples up to the above mentioned temperatures.

$\text{Al}_2\text{O}_3$  -  $\text{P}_2\text{O}_5$  system (samples 2, 3, 4 according to table). Samples of the system were thermally treated at 300 $^{\circ}\text{C}$  (2). Due to the fact that there had been  $\text{Al}_2\text{O}_3$  in excess

in the system (mole ratio  $\text{Al}_2\text{O}_3 : \text{H}_3\text{PO}_4 = 5:1$ ) aluminum oxide did not reactfully with orthophosphoric acid. It is shown that the greatest part of the samples consists of  $\text{Al}_2\text{O}_3$  bound with orthophosphates. There appear absorption lines on the IR spectra with frequencies  $1150, 1120, 1085, 800, 747, 725 \text{ cm}^{-1}$  and others that should be considered as valency and deformation fluctuations  $\nu(\text{Al-O}), \nu(\text{P-O})$  and  $\delta(\text{O-P-O})$  of groups characteristic of  $\text{AlPO}_4$  combinations. At least part of the above mentioned frequencies is proof of new connections appearing in the composition of the binder conditioned by the interaction of  $\text{Al}_2\text{O}_3$  with  $\text{H}_3\text{PO}_4$ . The fact is proved by the data of X-ray PD analysis: diffractogram reveals formation of  $\text{AlPO}_4$  ( $^\circ$ ) according to ASTM 11-500 (curves 2, Fig. 3, 4). This formation can be a different form of  $\text{AlPO}_4$  (bands of phosphate according to ASTM 20-45 are clearly visible, traces of phosphate according to ASTM 20-44). As is known [25]  $\alpha$  -  $\text{Al}_2\text{O}_3$  starts reacting with  $\text{H}_3\text{PO}_4$  at  $200\text{--}250^\circ\text{C}$ , hydrocomplexes Al of the  $\text{Al}_2(\text{OH})_3\text{PO}_4$ ,  $\text{Al}_3(\text{OH})_6\text{PO}_4$  type being formed therein. IR spectrum (2) reveals an absorption line with frequency  $3400 \text{ cm}^{-1}$  and (curve 2, Fig. 1). It may as well represent a band of the hydrophosphates of the OH-group. However O-H-zones in  $3200\text{--}3500 \text{ cm}^{-1}$  and  $1600\text{--}1650 \text{ cm}^{-1}$  are revealed practically in the spectra of all samples (see Table and Fig. 2), in all probability this band belongs to adsorption water.

To determine the influence of processing temperature on the physicochemical properties of the system, samples were analysed at  $900$  and  $1500^\circ\text{C}$  (compositions 3 and 4 in Tables).

The contour (3) of IR spectrum (curve 3, Fig. 1) does not reveal marked differences from spectrum (2), the fact testifies to minor structural modifications; probably there was a redistribution of binding in valency and deformation oscillations  $\nu(\text{Al-O}), \nu(\text{p-O}), \delta(\text{Al-O-P}), \nu(\text{P-O-P})$  etc in  $\text{AlPO}_4$  which results in consolidation of structural fragments and increase of strength in the system. X-ray analysis (3) reveals (curves 3, Fig. 3, 4) an increase of intensity of X-ray reflexes  $\text{AlPO}_4$  ( $^\circ$ ) (ASTM 11-500). It is visible on peak  $0.41 \text{ nm}$  as compared with the peak at (2). There appear reflexes of orthophosphates of other forms  $0.432; 0.386 \text{ nm}$  ( $\blacktriangledown$ ) (ASTM 20-45 and 20-44). Quantitative change of the initial phosphate  $\text{Al}_2\text{O}_3$  ( $\cdot$ ) and the one formed ( $^\circ$ ) is shown in Fig. 3 and 4 (curves 2 and 3). Thermal treatment at  $1500^\circ\text{C}$  (4) results in

further consolidation of binding  $\nu(\text{Al-O})$ ,  $\nu(\text{P-O})$ ,  $\delta(\text{Al-O-P})$  and others (curve 4, Fig. 1). It is especially noticeable in the range of  $\sim 800$  and  $\sim 450 \text{ cm}^{-1}$  X-ray diffraction testifies (curves 4, Fig. 3, 4) to the formation of  $\text{AlPO}_4$  ( $\circ$ ) with a high intensity reflex at 0.4103 nm. With the increase of intensity reflexes  $\text{AlPO}_4$  ( $\circ$ ) are preserved at 0.317; 0.287; 0.25; 0.1955; 0.163 nm. At 0.1894 nm a weak reflex of  $\text{AlPO}_4$  ( $\circ$ ) appears.

Composition of electrocorundum-  $\text{Al}_2\text{O}_3$  -  $\text{P}_2\text{O}_5$  (samples 5, 6, 7) (compounds by the table). The study of the binding system reveals the following:

In IR spectra of the composition thermally processed at  $300^\circ\text{C}$  (5) (curve 5, Fig. 1) absorption bands appear relating to valency and deformation oscillations  $\nu(\text{Al-O-Al})$ ,  $\delta(\text{Al-O-Al})$  and also  $\nu(\text{P-O})$  and  $\delta(\text{P-O-P})$  at 1160, 1100, 1020, 805, 780, 749, 720, 650, 624,  $592 \text{ cm}^{-1}$  etc, that refer to the combination  $\text{AlPO}_4$ , the fact is confirmed by the peaks on diffractograms (curves 5, Fig. 3, 4) at 0,41; 0,308; 0,19 nm ( $\circ$ ) (ASTM 11-500); of weak intensity peaks 0,438; 0,422; 0,381 nm according to ASTM 20-45 and 20-44 they correspond to other modifications of  $\text{AlPO}_4$ . Rather intensive peaks of aluminum oxide are preserved at 0,348; 0,209; 0,174; 0,16; 0,1403; 0,1374 nm.

Thermal treatment at  $900^\circ\text{C}$  (6) brings about the formation of the same phosphates as in system (3) but with more definite bands of absorption at 800, 780, 759, 644, 588, 515, 460,  $396 \text{ cm}^{-1}$ , etc (curve 6, Fig. 1). It testifies to establishment of a frame structure. X-ray plates (curves 6, Fig. 3 and 4) of the composition reveal new X-ray reflexes 0.41; 0.318; 0.288; 0.250; 0.163 nm, referring to  $\text{AlPO}_4$  ( $\circ$ ) (ASTM 11-500). Lines of other forms of  $\text{AlPO}_4$  ( $\nabla$ ) (ASTM 20-45 and 20-44) have been detected with significances 0,432; 0,386, as well as weak lines  $\text{SiO}_2$  ( $\diamond$ ) which accompany initial components.

The IR spectrum over  $1500^\circ\text{C}$  (curve 7, Fig. 1) is characterized by absorption bands with the significances of frequencies close to frequencies (5) and (6). However modification of intensities of frequencies in the range of  $1100 \text{ cm}^{-1}$  and  $800\text{-}350 \text{ cm}^{-1}$  relating to  $\text{AlPO}_4$  testify to stabilization of structural parameters of the given composition. The X-ray analysis data confirm these suppositions (curves 7, Fig. 3 and 4). An increase of intensity however is observed in peaks 0.407; 0.318; 0.287; 0.2506; 0.196 nm  $\text{AlPO}_4$  as compared to (5) and (6). Other phosphates are not

detected. The quantity of  $\text{SiO}_2$  ( $\diamond$ ) remained without modifications (0,424; 0,333; 0,181 nm).

Composition of electrocorundum-  $\text{Al}_2\text{O}_3$  - PB after deformation at creeping (compounds by the table). The physicochemical properties of the composition after deformation under standard load of 0,2 MPa and creeping at different temperatures, loads and curing time are studied on samples of compositions 8, 9, 10, 11, 12.

Sample 8 is used to study the effect of deformation under the load of 0,2 MPa on the processes of structural changes occurring in the composition at temperature of  $1450^\circ\text{C}$ . Comparison of UR spectrum (8) (curve 8, Fig. 2) with the spectra (5), (6), (7) testifies to minor modifications in intensity of absorption bands in phosphates only, and to emerging of new absorption bands with frequencies  $655$  and  $451\text{cm}^{-1}$ , relating to oscillations  $\delta(\text{P-O-P})$  and  $\nu(\text{M-O})$  berlinite and crystaballite ( $\text{AlPO}_4$ ) [20].

X-ray plate (8) reveals (curves 8, Fig. 5, 6) lines 0.406; 0.318; 0.287; 0.25; 0.1955; 0.188 nm, relating to  $\text{AlPO}_4$  ( $\circ$ ) (ASTM 11-500), and also lines at 0,43; 0,6383 nm referred to other forms of phosphates ( $\blacktriangledown$ ) (ASTM 20-45 and 20-44) that by their intensity exceed the intensity of similar peaks at (7). The peaks 0.424; 0.333; 0.245; 0.227; 0.2126; 0.1815; 0.154 nm  $\text{SiO}_2$  ( $\diamond$ ) are preserved. Decrease of intensity of lines  $\text{Al}_2\text{O}_3$  is observed therein.

In IR spectrum of the composition after  $1300^\circ\text{C}$  (9) there is no absorption band  $\delta(\text{O-H})$  in the range  $\sim 1600\text{ cm}^{-1}$  ( $\text{P-OH}$ ), and absorption bands relating to binding  $\text{P-O-P}$  characteristic for high-temperature phosphates have appeared. By the intensity of X-ray peaks their quantity equals that of in (9) and (8), however it is greater in (9) than in (7). Samples (9) and (8) also contain approximately equal quantity of phosphates, however they are more in (9) than in (7) due to the difference in temperatures and curing time under load (curves 7, 8, 9, Fig. 5, 6).

In IR spectrum (10) at the increase of temperature up to  $1400^\circ\text{C}$  modification of the forms of absorption bands (curve 10, Fig. 2), certain displacement of frequencies, modification of intensity in the range of  $800\text{-}350\text{ cm}^{-1}$  is marked. Decrease of intensity of the absorption band at  $1092\text{ cm}^{-1}$ , which is inherent in compositions containing the binder  $\text{P-O}$ , is observed under the effect of high temperatures. The fact of a decrease in intensity of bands in compounds with the  $\text{P-O}$  binding is also marked by other authors

[22, 23]. A decrease in the quantity of phosphate of the given sample is observed on the X-ray PD (curve 10, Fig. 5 and 6). It is conditioned by the structural transformations occurring in the compositions due to their creeping within 28,5 hours.

The IR spectrum of sample 11 (curve 11, Fig. 2) is characterized by a modification in the contour of absorption bands in the range of  $800\text{--}350\text{ cm}^{-1}$  as compared to IR spectra (4, 9, 10). The band at  $990\text{ cm}^{-1}$  conditions the presence of bindings P-O-P and P-O, a decrease of their intensity testifies to a decrease of the quantity of phosphates and formation of other (high temperature) modifications of phosphates, crystaballite among them [22, 23]. By the method of differential thermal analysis (DTA) transition of  $\text{AlPO}_4$  (berlinite)  $\rightarrow 1230^\circ \rightarrow \text{AlPO}_4$  (crystaballite) is observed in the system  $\text{Al}_2\text{O}_3 - \text{P}_2\text{O}_5$ . Comparison diffractograms of the composition (11) (curves 11, Fig. 5 and 6) with (4), (9), (10) also testifies to a decrease in the quantity of phosphates at (11), the latter fact being connected with deformation processes at creeping.

Sample 12 is obtained with the use of ACPB. In IR spectra new bands appeared  $698, 645, 430\text{ cm}^{-1}$  relating to  $\delta(\text{P-O-P})$ ,  $\nu(\text{M-O})$  that refer evidently to chromium phosphate. The IR contour testifies to the stability of the composition. Not very pronounced reflexes  $0.4087; 0.328; 0.288; 0.25; 0.1955\text{ nm AlPO}_4$  ( $^\circ$ ) (ASTM 11-500) are discerned on the X-ray PD (curves 12, Fig. 5 and 6), as well as weak lines  $0.4219; 0.3329; 0.1812\text{ nm SiO}_2$  ( $\diamond$ ) that create the frame of the composition cementing the particles of the filling.

#### Petrographic analysis

The analysis is conducted with the help of microscope MIN-8 in immersion liquid with 320x magnifying of samples without deformation (7) and after deformation at creeping, the temperature being  $1500^\circ\text{C}$  (11). The sample (11) when magnified clearly reveals angular birefracting grains (Fig. 7), the size of the largest grain being not more than 0,1 mm in diameter. The indices of refractions of grains are very high (over 1, 7) and birefracton equals 0.009.

By some data all these grains are represented by corundum, the fact is also confirmed by diffractometric data. The smallest grains are of the size not more than 0,003-0,005 mm. Among the grains there is contained a great amount (about 30-50% of



the sample volume) of very thin dust consisting of tiniest (up to 0,001 mm) grains, the optical constants of which (indices of refraction, birefracton, etc) do not lend themselves to any determination even at the greatest magnifying power of the microscope. Only by the X-ray PD data it is possible to surmise that they represent aluminum phosphate, a part of them being finely grated alumina. More often than not these finest crystallites (grains) form semi-translucent round balls, white in the reflected light. This thin dispersion mass stipulates the mechanism of high temperature creeping. In (7) the quantity of phosphates formed per unit of area in the sample is higher than in (11), and the distances between particles of new formations, inert fillings are closer, the fact also being connected with the deformation of the composition.

The method of IR spectroscopy, X-ray PD and pectrographic analysis is used to study the binding systems and compositions on the basis of aluminum oxide and phosphate binding ( $H_3PO_4$  or ACPB). The influence of the compounds in the composition, the type of the binder and the temperature of processing is established, as well as the test condicions of the composition at creeping and their effect on the structural transformations and behaviour at high temperatures.

The compositions as the result of a long process of deformation at high temperatures and loads reveal modifications of physicochemical properties of the materials, and as a consequence a change in their structures. In all probability it determines the mechanism of the creeping process that is caused by the change of the matrix (frame) cementing the grains of electrocorundum in the composition: electrocorundum- $Al_2O_3$ - phosphate binder (PB).

## REFERENCES

1. A.G.Evans, T.G. Langdon, Structural Ceramics. Metalurgy, ed. (Moscow, 1980), p. 160
2. G.N.Maslennikova, R.A.Mamaladse, S. Midzuta and K.Koumoto. Ceramic Materials. Stroiizdat, ed (Moscow, 1991), p. 96
3. V.S. Bakunov. Russian Journal of Refractory Materials. 12: 2 (1997)
4. D.N. Poluboiarinov, V.S. Bakunov. Russian Journal of Inorganic Materials. 3: 374 (1965)
5. V.S. Bakunov, A.V.Beliakov. Russian Journal of Inorganic Materials. 32: 243 (1996)
6. V.S. Bakunov, A.V.Beliakov. Russian Journal of Inorganic Materials. 33: 1553 (1997)
7. E.M.Grishpun, Yu.E.Pivinskii, E.B.Rojkov, D.A.Dobrodon, I.A.Galenko, T.N. Kononova Russian Journal of Refractory Materials and Technical Ceramics. 3: 37 (2000)
8. L.G.Sudaskas. Russian Journal Cement and its application 2-3: 34 (1999)
9. K.Nakamoto. IR Spectra and CD Spectra of Inorganic and co-ordination compounds. Mir, ed. (Moscow, 1991)p. 283
10. K.Nakamoto. Infrared Spectra of Inorganic and co-ordination compounds. Mir, ed. (Moscow, 1966), p. 145
11. V.V.Pechkovsky, R.Ia. Melnikova, E.D.Dziuba, I.I.Barannikova, V.B.Nikanovich. The Atlas of the Phosphate IR Spectra.Ortophosphates. Nauka, ed. (Moscow, 1981), p. 143
12. I.V. Tananaev. The Atlas of the IR Spectra of Phosphates. Condensated Phosphates. Nauka, ed. (Moscow, 1985), p. 114
13. A.N.Lazarev, A.P.Mirgorodsky, I.S.Ignatiev. The vibration spectra of complecated oxides. Nauka, ed. (Leningrad, 1975), p. 229
14. E.N.Yurchenko. Modern Vibrational Spectroskopy of Inorganic Compounds. Nauka, ed. (Novosibirsk, 1990), p. 230
15. Leo D. Fredrickson, Jr. Anal. Chem. 26: 1883 (1954)
16. John M. Hunt, Mary P. Jr. Anal. Chem. 22: 1478 (1950)

17. U.Sh.Shayachmetov. The Compositions Materials on basis of  $\text{Si}_3\text{N}_4$  and phosphate binders. Intermet Inginiring, ed. (Moscow, 1999), p.17
18. A.A. Pashcenko. New Cements. Budivelnik, ed. (Kiev, 1979), p. 139
19. James Alamo and Rustum Roy. Comm. Amer. Ceram. Soc. 80 (1984)
20. Francisco J. Gonzales and John Halloran. Amer. Ceram. Soc. Bull. 59. 727 (1980)
21. U.Sh.Shayachmetov, I.M.Valeev, K.A.Vasin, R.S.Malikov. Russian Journal of Refractory Materials and Technical Ceramics. 11: 21 (1999)
22. V.V.Gerasimov. Inorganic Polimer Materials on the basis of  $\text{SiO}_2$  and Phosphorous, Stroiizdat, ed. (Moscow, 1993) p. 131
23. B.A.Kopeikin. The Technology and Properties of phosphate Materials. Stroiizdat, ed. (Moscow, 1974) p. 17
24. L.L.Vaniceva, O.V. Efremova, Iu.B.Materkin, S.P.Shmitt - Fogelevich. The Investigation and Application Methods of Refractory Materials in Metallurgy. (Moscow, 1983) p. 44
25. John R. Van Wazer. Phosphorus and its Compounds. Inlit, ed. (Moscow, 1962), p.26

Table

Compositions tested and relating of main oscillating frequencies ( $\text{cm}^{-1}$ ) revealed in IR spectra of absorption in samples on the basis  
of aluminum oxide and phosphate bindings\*  
(band intensity: s-strong, m. - middle, w. -weak, wi- wide, b - berding)

N <sup>o</sup> of sa m pl e	Compositions	$\nu(\text{OH})$ $\nu_{\text{as}}(\text{OH})$ , $\nu_{\text{s}}(\text{OH})$ ( $\text{Al}_2\text{O}_3$ )	$\delta(\text{OH})$ M-OH $\text{H}_2\text{O}$ ( $\text{Al}_2\text{O}_3$ )	$\nu(\text{Al}-\text{O}-\text{Al})$ , $\nu(\text{O}-\text{Al}-\text{O})$ , (P-O) ( $\text{AlPO}_4$ ) berlinite (crystaballite)	$\text{Al}_2\text{O}_3$	$\nu(\text{P}-\text{O}-\text{P})$	$\delta(\text{Al}-\text{O}-\text{Al})$ $\delta(\text{P}-\text{O}-\text{P})$ ( $\text{Al}_2\text{O}_3$ )	$\text{AlPO}_4$ $\delta(\text{P}-\text{O}-\text{P})$	$\delta(\text{Al}-\text{O}-\text{Al})$ ( $\text{Al}_2\text{O}_3$ )	$\nu(\text{M}-\text{O})$ $\text{AlPO}_4$ (berlinite) ( $\text{Al}_2\text{O}_3$ )	$[\text{PO}_4]^{3-}$	$\text{AlPO}_4$ ( $\text{Al}_2\text{O}_3$ )	$[\text{PO}_4]^{3-}$
1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	$\alpha - \text{Al}_2\text{O}_3$	(3390 w. wi.)	(1595 w.wi)	(1090 s. wi.)	(780 b.)	-	(720 b.)	(639 m. )	(590 s.)	( 448 m.)	-	(393 w.)	-
2	$\alpha - \text{Al}_2\text{O}_3$ (100 %), $\text{H}_3\text{PO}_4$ (24 %) $T_0 = 300^\circ\text{C}$	3400 w. wi.	1635 w. wi.	1150b., 1120b., 1070b.	800 b.	747 b.	725 b.	648 w. 614 w. -	511 b.	460 s. 450 s	400 b.	390 m. 384 w.	348 w.
3	same composition $T_0 = 900^\circ\text{C}$	3400 w.wi.	1600 w.wi.	1170s., 1120 b., 1030 b.	800 b.	752 b.	730 b.	648 w. 644 b	510 b	470 s 465 s	405 b	392w -	348w 324b
4	$\alpha - \text{Al}_2\text{O}_3$ (100 %), $\text{H}_3\text{PO}_4$ (24 %) $T_0 = 1500^\circ\text{C}$	3405 w.wi.	1630 w.wi.	1190 b., 1120s., 1080 b.	800 b.	755 b.	728 w.	- 628 b	544 b	476 w	403 b	392 w 384 w	348w.
5	e/c N <sup>o</sup> 50 -35% e/c N <sup>o</sup> 6 - 40% FGAN ( $\text{Al}_2\text{O}_3$ )- 25% $\text{H}_3\text{PO}_4$ - 12%, $T_0=300^\circ\text{C}$	3400 w.wi.	1650 w.wi.	1160 b. , 1100 s.wi, 1020 b	805 m. 780 m.	749 b.	720 w.	650 w 624 w 592 w	515w	460w	?	380w.	340w.
6	same composition $T_0 = 900^\circ\text{C}$	3405 w.	1630 w.wi.	1150 b., 1096 s.wi., 1020 b.	800 m. 780 m.	759 w.	725 w.	644 w - 588 m	515 w	460 m	396w	370 b.	348 w.
7	e/c N <sup>o</sup> 50 -35% e/c N <sup>o</sup> 6 - 40% FGAN - 25 % $\text{H}_3\text{PO}_4$ - 12%, $T_0 = 1500^\circ\text{C}$	3520 w.wi. 3410 w.wi.	-	1150 b., 1108 s. wi., 1060 b	800 b. 782 m.	748 w.	722 w.	646 w 604 w -	512 m	464 m	398w	388w. -	340 w.
8	Composition analogous to 5. Deformation under load 0,2Mpa by Standart 4070-83, max.t.- $1450^\circ\text{C}$	3412 w.wi.	1640w.wi.	1170 b., 1100 s.wi., 1040 b.	802 m. 782 m.	760 w.	731 w.	655 m. 645 m. 599 m.	512 b.	461 m. 451 m.	398 w.	386 w. 372 w.	350w.

1	2	3	4	5	6	7	8	9	10	11	12	13	14
9	same composition T = 1300 °C $\tau$ = 159,54 hr $\sigma$ = 0,6 MPa	3400 w.wi.	–	1115 b., 1092 s., 1045 b.	798 m. 779 m.	750 b.	738 w.	695 w. 680 w. 646 m. 610 w. 590 m.	515 b.	460 m. 455 b. 420 b.	396 w.	388 w. 370 w.	348 w. 304 w.
10	same composition T = 1400 °C $\tau$ = 28,50hr $\sigma$ = 0,6MPa	3405 w.wi.	1660 w.wi.	1160 b., 1090 s.wi., 1025 b.	805 b. 782 m.	760 b.	730 w.	690 w. 660 b. 600 w. 590 w. 565 w. 532 b.	512 b.	465 m. 453 m. 430 b.	397 w. 388 m.	372 w.	344 w. 328 w. 314 w.
11	same composition T = 1500°C $\tau$ = 57 hr $\sigma$ = 0,6 MPa	3540 w.wi. 3400 w.wi.	1655 w.wi. 1610 w.wi.	1150 b., 1085 s., 990 b.	805 m. 783 m.	751 b.	735 w.	696 w. 650 m. 604 w. 590 m. ?	512 w.	462 m. 435 m.	397 m.	388 m. 372 w.	345 w. 326 w. 312 b.
12	same composition ACPB instead of H <sub>3</sub> PO <sub>4</sub> T <sub>o</sub> = 300 °C T = 1200 °C, $\tau$ = 47,40 hr $\sigma$ = 0,6 MPa	3404 w.wi.	–	1110 b., 1091s., 1062 b.	798 m. 780 m.	743 b.	722 w.	698 b. 670 w. 645 m. 610 w. 590 m.	514 w.	462 m. - 430 b.	398 w.	388 w. 372 w.	356w.

\* Notations: e/c - electrocorundum; FGAN - finely grated alumina, nonhydrated; ACPB - aluminumchromphosphate binder; T<sub>o</sub> - temperature of treatment; T - temperature of creeping test;  $\tau$  - time of creeping test;  $\sigma$  -load at creeping

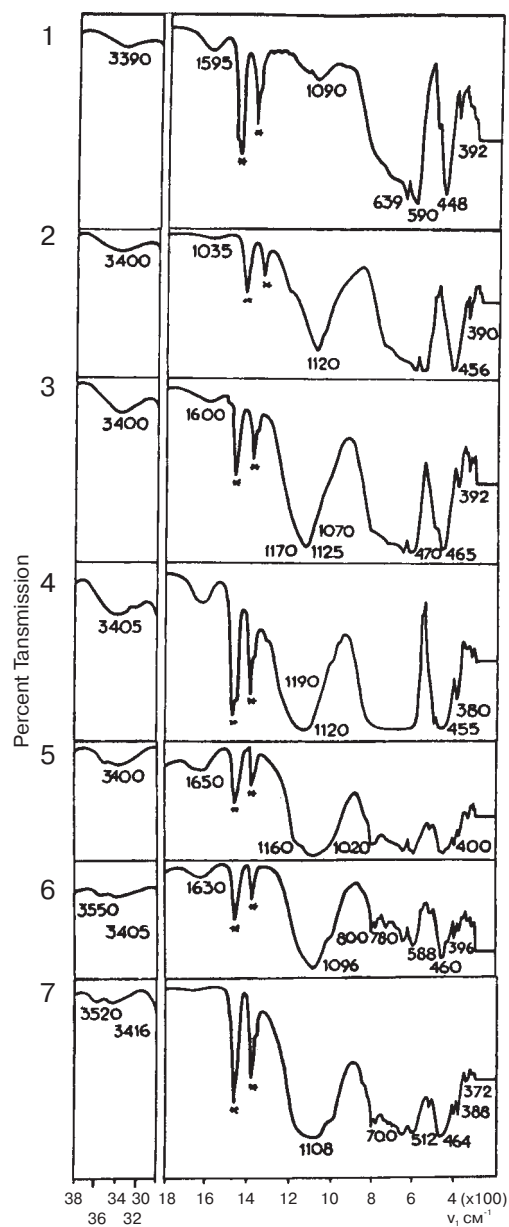


Fig. 1

IR spectra of absorption  $\text{Al}_2\text{O}_3$ , systems  $\text{Al}_2\text{O}_3\text{-P}_2\text{O}_5$  and compositions (according to the table) after thermal treatment at temperatures,  $^{\circ}\text{C}$  : 2 and 5 - 300, 3 and 6-900, 4 and 7 - 1500.

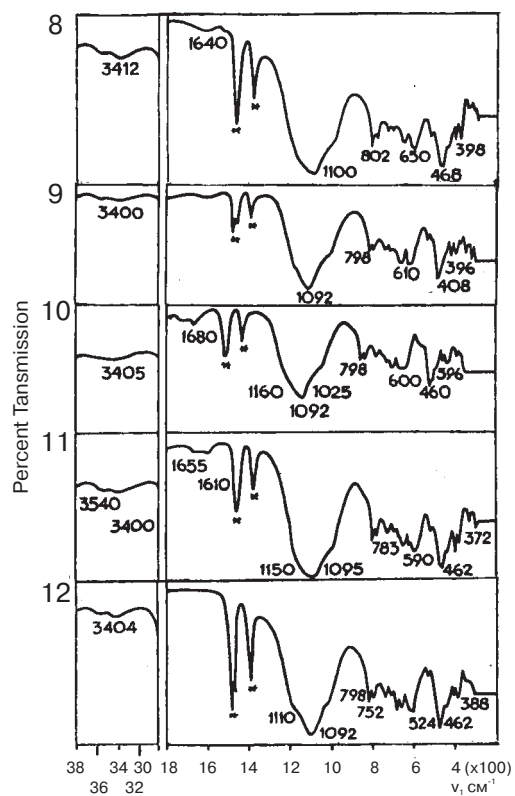


Fig.2

IR spectra of absorption of compositions (according to the table) after deformation under load of 0,2 MPa and with creeping at temperatures, °C : 8 - 1450,9 - 1300, 10 - 1400, 11 - 1500, 12 - 1200.

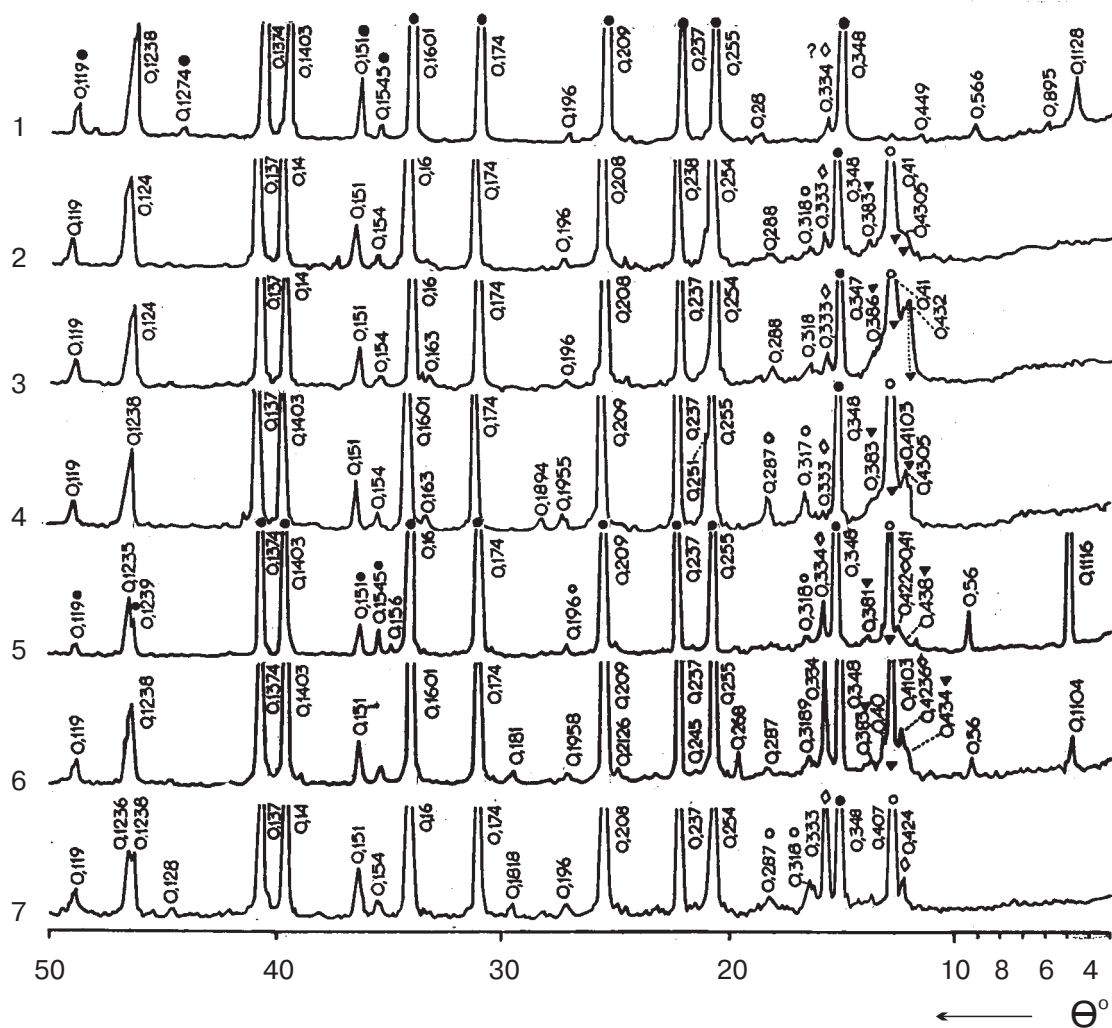


Fig.3

$\text{Al}_2\text{O}_3$ , systems  $\text{Al}_2\text{O}_3 - \text{P}_2\text{O}_5$  and compositions (according the table) after thermal treatment at temperatures, °C: 2 and 5 - 300, 3 and 6 - 900, 4 and 7 - 1500. Notation of relating the main peaks: ● -  $\alpha\text{-Al}_2\text{O}_3$ ; ○ -  $\text{AlPO}_4$  - phosphate; ▼ - other phosphates; ◇ -  $\alpha\text{-SiO}_2$  quartz.



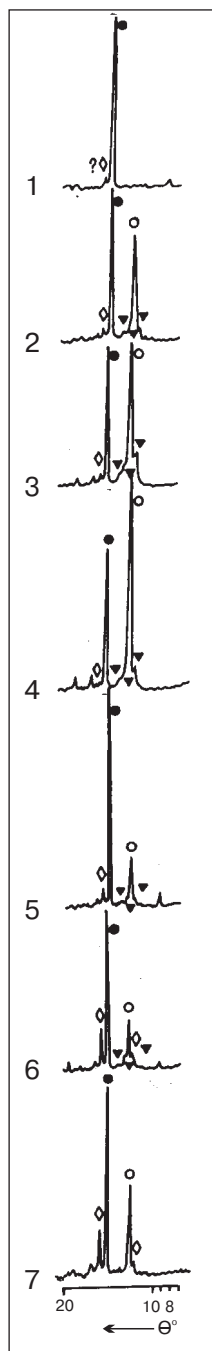


Fig. 4

Fragments of diffraction from fig. 3  $\text{Al}_2\text{O}_3$ , system  $\text{Al}_2\text{O}_3 - \text{P}_2\text{O}_5$  and compositions (according to the table) after thermal at temperatures, °C: 2 and 5 - 300, 3 and 6 - 900, 4 and 7 - 1500. Notation of relating the main peaks:  
 ● -  $\alpha\text{-Al}_2\text{O}_3$ ; ○ -  $\text{AlPO}_4$  - phosphate; ▼ - other phosphates; ◇ -  $\alpha\text{-SiO}_2$  quartz.

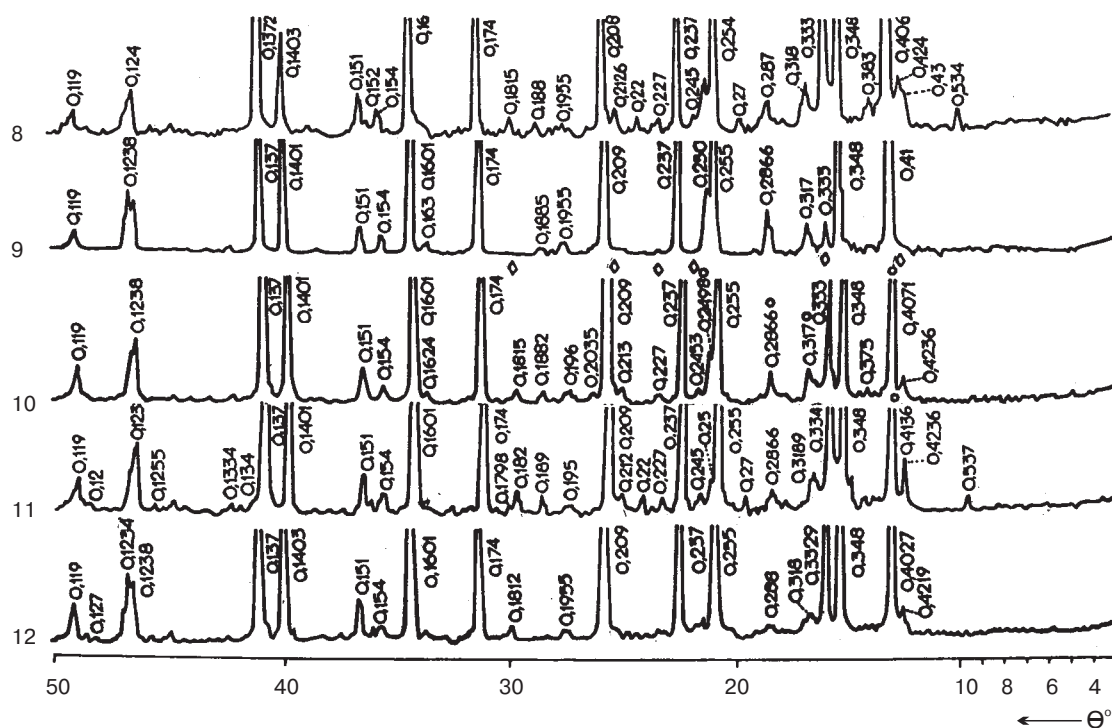


Fig.5

Diffraction of compositions (according to the table) after deformation under load 0,2 MPa and creeping at temperatures, °C : 8 - 1450, 9 - 1300, 10 - 1400, 11 - 1500, 12 - 1200. Notation of relating the main peaks: ●- $\alpha$ -  $\text{Al}_2\text{O}_3$ ; ○-  $\text{AlPO}_4$  - phosphate; ▼- other phosphates; ◇- $\alpha$ -  $\text{SiO}_2$  quartz.

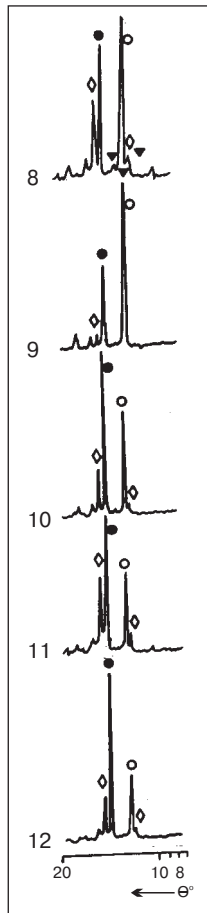


Fig.6

Fragments of diffraction from Fig. 5 (according to the table) after deformation under load 0,2 MPa and creeping at temperatures, °C : 8 - 1450, 9 - 1300, 10 - 1400, 11 - 1500, 12 - 1200. Notation of relating the main peaks:  
 ● -  $\alpha$ -  $\text{Al}_2\text{O}_3$ ; ○ -  $\text{AlPO}_4$  - phosphate; ▼ - other phosphates; ◇ -  $\alpha$ -  $\text{SiO}_2$  quartz.

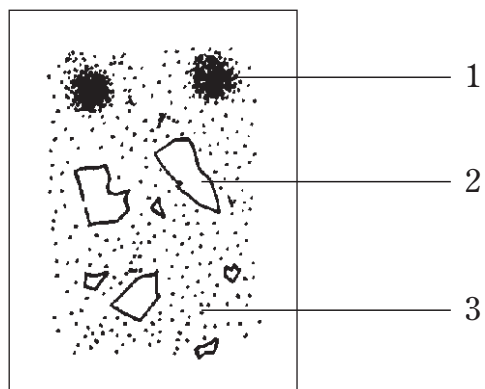


Fig.7

Sample 4 in immersion liquid after deformation at creeping at 1500<sup>0</sup>C.

- 1 - Congestion of submicroscopic grains of the cementing mass.
- 2 - Large crystals from electrocorundum.
- 3 - Thin dispersion mass consisting by X-ray data of aluminum phosphate.